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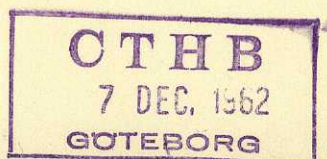
ELECTROMAGNETIC SEPARATION
OF ISOTOPES IN A SMALL LABORATORY
MACHINE

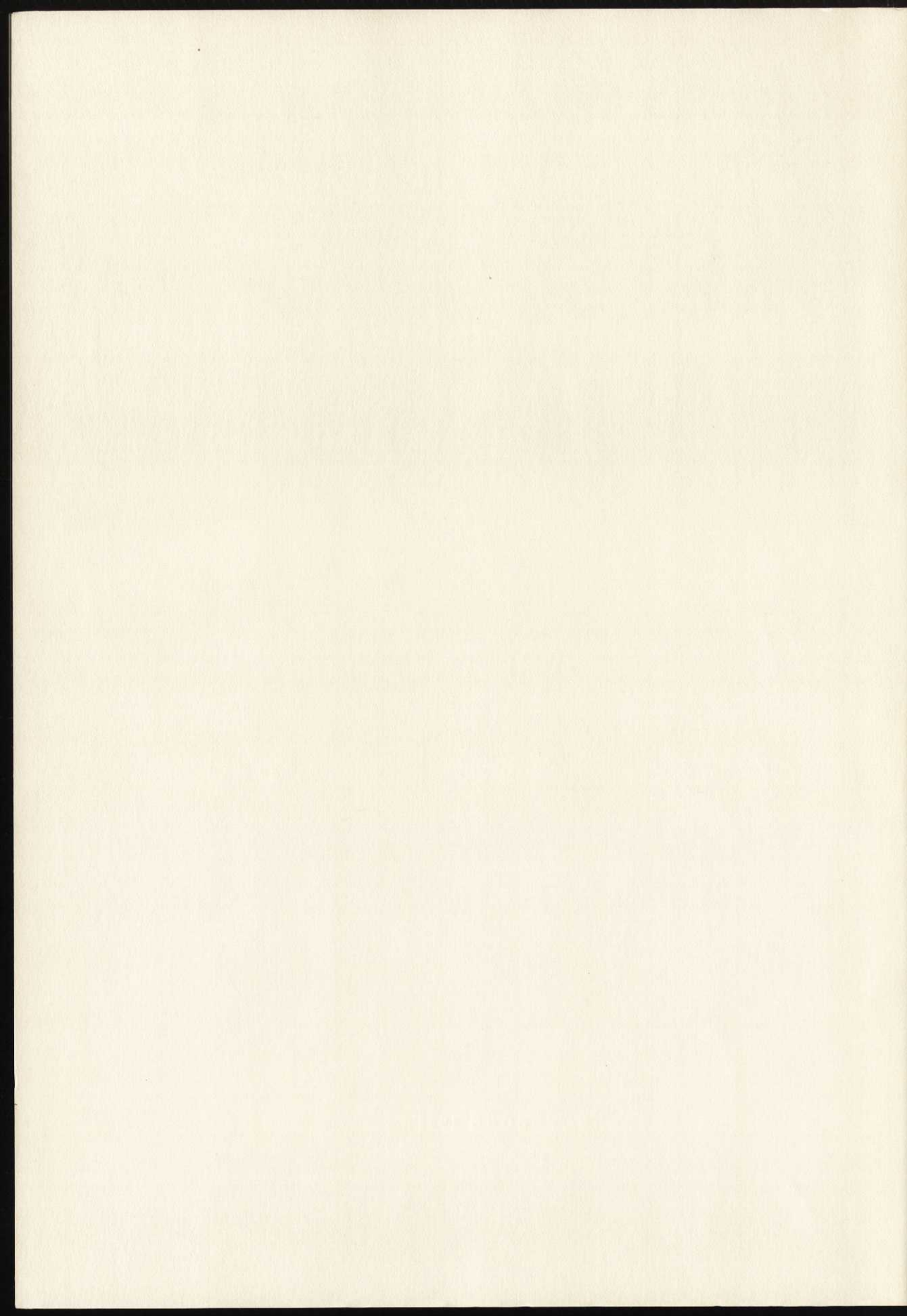
by

O. ALMÉN



GÖTEBORG 1962





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AKADEMISK AVHANDLING

SOM MED TILLSTÅND AV CHALMERS TEKNISKA HÖGSKOLA
FÖR TEKNOLOGIE DOKTORSGRADS VINNANDE TILL
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ELANDERS BOKTRYCKERI AKTIEBOLAG
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This thesis includes the following papers:

- I. O. ALMÉN, G. BRUCE and A. LUNDÉN, Electromagnetic isotope separator in Gothenburg, Nuclear Instruments 2 (1958) 249-260.
- II. O. ALMÉN and K. O. NIELSEN, Systematic investigation of a magnetic ion source for an electromagnetic isotope separator, Nuclear Instruments 1 (1957) 302-322.
- III. O. ALMÉN and G. BRUCE, Collection and sputtering experiments with noble gas ions, Nuclear Instruments and Methods 11 (1961) 257-278.
- IV. O. ALMÉN and G. BRUCE, Sputtering experiments in the high energy region, Nuclear Instruments and Methods 11 (1961) 279-289.
- V. Electromagnetic separation of isotopes in a small laboratory machine.

Summary

An electromagnetic isotope separator is principally a large mass spectrometer. An ion beam of positive, singly charged particles with an energy of a few keV passes a homogeneous magnetic field. Because of the combined effect of the ion velocity and the magnetic field the beam is forced to follow a circular path through the magnet. At the same time it is split up in different beams, one for each of the different ion masses present in the original beam. In this way the separation of isotopes is made possible.

The isotope separator in Gothenburg is a 90° sector machine and is described in paper I. The magnet has a mean radius of 100 cm and the acceleration voltage is maximum 50 kV. These data mean that an ion current of from 10 to several hundred μA , depending on the ion mass, can be processed and isotopes collected about 10 mm apart for mass number 100, at a line half width of 1–3 mm. In order to reach a high isotopic enrichment, facilities for stabilisation of both the magnetic field and the high tension have been applied.

The overall performance of the isotope separator is very dependent on the ion source. Therefore a lot of work has been devoted to development of suitably constructed ion sources and to investigation of their operation. Paper II contains a systematic study of the influence on ion current and resolving power from different parameters inside the ion source and in the acceleration gap. It was proved that the focusing conditions are mainly determined by the shape of the plasma boundary at the outlet of the ion source, this means determined by the ion density inside the source. Only a small adjustment of the focusing can be obtained by varying the lens potential of the electrostatic lens system. The results obtained in paper II have been very valuable for the further use of this type of ion source.

An important part of the separation process is the collection of ions for use in various types of other research apparatus. The direct collection method has mostly been applied in small laboratory separators. This means that the ion beam at 30–50 keV energy hits a metal target directly and because of the penetration of atomic particles

with high velocity, ions can be trapped in the metal lattice. Usually only very small quantities can be collected by this method, but in the case of noble gas ions, it is the only possible collection method. In paper III saturation of different noble gases in various backing materials has been measured. The amounts collected vary systematically within the periods of the periodic system of elements. A few of the phenomena causing a saturation have also been investigated. E.g. the sputtering process is of great importance for the collection of isotopes and it also varies periodically through the periodic system. Sputtering measurements are also of interest from other points of view, e.g., ion propulsion has been discussed for future space flights. Here sputtering must be taken into account, since it puts a limit to the lifetime of the ion machines. Although papers III and IV were intended as a study of collection phenomena only, both have received a stimulating interest from people working with other aspects of the sputtering process. At a number of conferences the results have already been referred to and discussed.

Since 1953, when the machine was first put into operation, a great number of samples of stable isotopes have been prepared for the electrostatic generator at the Physics Division and for other groups in Sweden and abroad, working with charged particle bombardment. The technique has been more and more refined, so nowadays preparation takes place with a retarded ion beam, sometimes hitting a very thin backing foil of a few $\mu\text{g}/\text{cm}^2$. An approximately known quantity can now be deposited in a thin homogeneous layer, which is very important for high precision work. In addition a number of radioactive samples have been prepared for beta-gamma spectroscopy in Sweden and abroad. Here the collection is quite easily performed, since the quantity involved is very small.

1. Introduction

Since 1934, when the first isotope separations were performed by M. L. Oliphant et al.¹⁾ and W. R. Smythe et al.²⁾ using mass spectrographs, the facilities for electromagnetic isotope separation have increased tremendously. The greatest development was of course that performed in the USA 1940—45, when a large number of calutrons were built for the production of uranium-235. However, most of this machinery is now broken up. Only two complete sets remain, reconstructed, one is utilized for stable isotope production, and part of the other is intended for separation of plutonium.³⁾ In 1957 the site and general data were compiled in paper I for about 18 machines and groups of machines known to be in operation at that time. Today the number of machines in use, or to be put into use in the near future, is around double this number. This rapid development indicates the applicability of electromagnetic isotope separators, especially the small machines — laboratory isotope separators.

A great number of these small machines are used for the preparation of radioactive samples for beta-gamma spectroscopy. Material produced in both reactors and cyclotrons is processed, and the obtained samples may be considered ideal for precision measurements. Another application is the preparation of thin targets of stable isotopes for charged particle bombardment, where nuclear reactions are studied. A limitation is that targets thicker than some ten $\mu\text{g}/\text{cm}^2$ are in most cases difficult to prepare in a laboratory machine, because of the long time necessary for operation or factors limiting the collection of the isotopes.

Many of the new isotope separators are of the 90° type for which a great deal of the development work has been done in Scandinavia by four groups working in close cooperation at Copenhagen, Stockholm, Upsala and Gothenburg. These four machines are quite similar and were completed during the period 1943—53. All four separators are described in the literature as well as the greater part of the development work on ion sources, arrangements for increased overall performance and investigations of collection phenomena.

The author has had the opportunity of taking an active part in this development work and the present paper presents some of the problems encountered using laboratory isotope separators. It is hoped that the present paper will be of use for separator groups now starting new machines, and who therefore will meet similar problems to those experienced by the author. For a thorough description of the machine itself the reader is referred to e.g. paper I, and in the following a few problems of current interest will be discussed in detail after a short general review.

2. 90° isotope separator

A 90° electromagnetic isotope separator is principally the same type of instrument as a mass spectrometer. Instead of a high resolution and good facilities for accurate measurements of isotopic abundances — main problems in a mass spectrometer — in an isotope separator the chief stress is laid upon a reasonably high ion current and methods for the efficient use of all kinds of ions. Nowadays the resolving power of a 90° isotope separator is comparable with that of a low or medium resolving mass spectrometer at an ion current of the order of 100 μ A.

Fig. 1 shows schematically a 90° isotope separator. An ion beam of positive ions is drawn out from an ion source, is accelerated to 30—50 keV and is passed through a 90° magnetic sector field. Here the ions are forced to follow circular paths which are determined by the mass, charge, velocity of the ions and by the magnetic field. The original beam contains mainly singly charged ions. In the magnetic field the beam is split up into different ion beams, representing each isotope of the element fed in to the ion source. In order to get these beams focused at the collector the ions must be accelerated to the same energy. The energy spread of ions leaving the ion source is only a few eV, while the accelerating voltage is many keV, so this condition is fulfilled.

After extraction from the source the beam passes through an electrostatic lens system containing three cylindrical electrodes (fig. 2). The first, called the extraction electrode, is normally connected to ground, but can also be connected to a negative voltage. The ion source is fed with a positive high tension which thus determines the ion energy. The beam shape is determined mainly at the exit from the source by parameters inside the source and in the gap between the source and the first electrode, the acceleration gap. The middle elec-

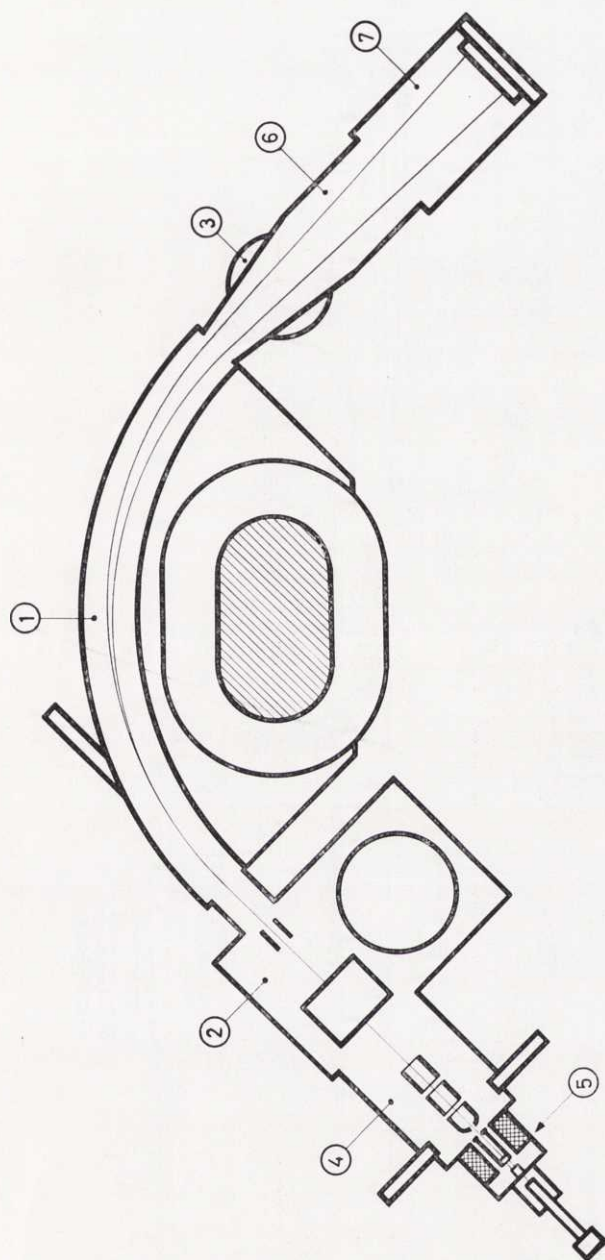


Fig. 1. Schematic diagram of the isotope separator. (1) magnet, (2), (3) vacuum valves and two diffusion pumps, (1500 l/s each), (4) acceleration chamber, (5) ion source, (6) extension chamber, (7) collection chamber.

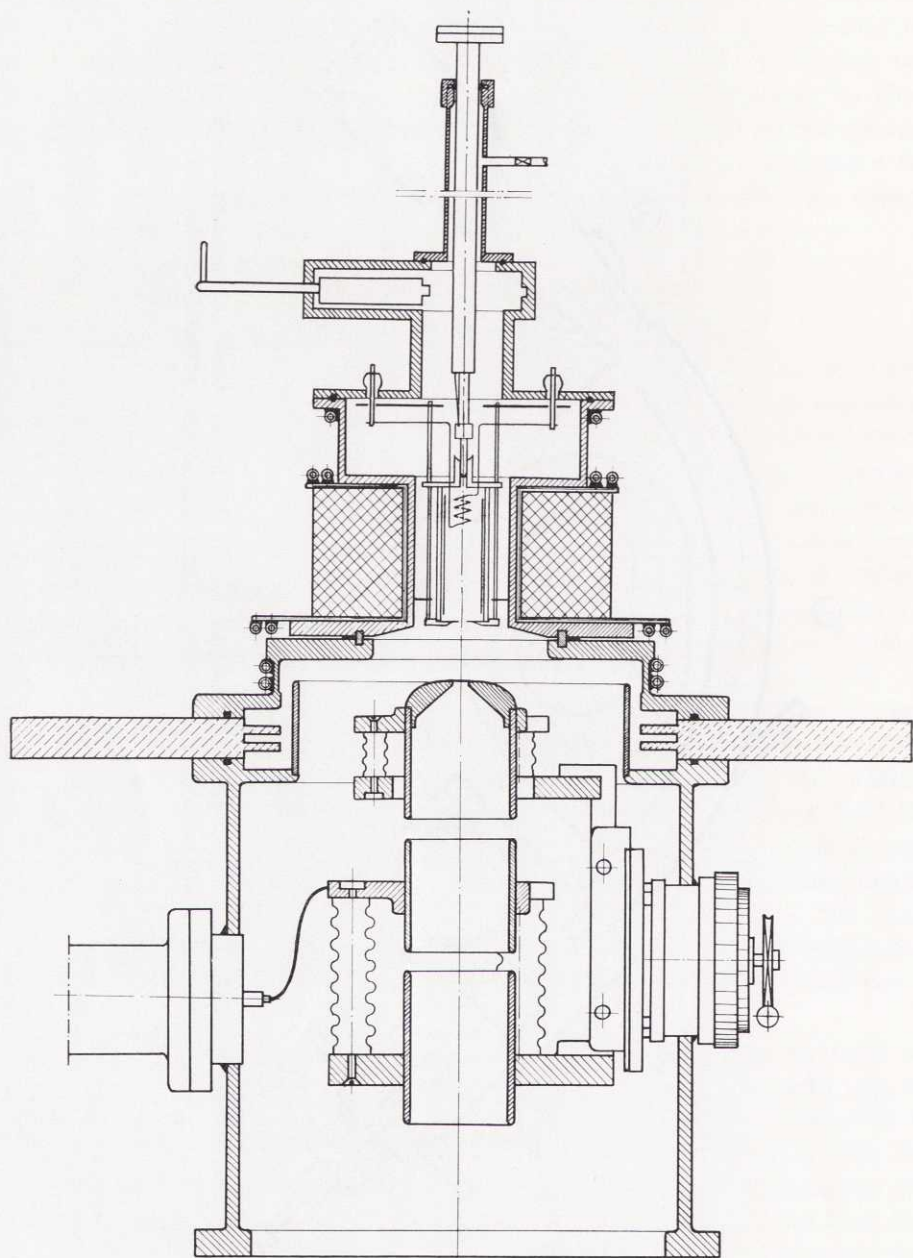


Fig. 2. Lens system and ion source. From right to left: ion source, extraction electrode at negative potential or ground, second electrode at lens potential, third electrode at ground. A perspex plate insulates the ion source from the acceleration chamber in which the lens system can be moved in two directions from the operators desk.

trode is connected to a positive potential, called the lens potential, and the third electrode is at ground. The complete set is an electrostatic lens which permits a change of the ion beam shape.

With the collector at a distance from the exit edge of the magnet the ion beam must enter the magnet slightly divergent. The 90° magnet focuses the isotopic beams in only one direction. Therefore we get lines at the collector parallel to the magnetic lines of force. In order to decrease the height of the lines a lens in the form of three pairs of plates is added between the lens system and the magnet. To the middle pair is applied a positive high tension. With this arrangement the lines can be compressed to about half their original height.

In the collection chamber the isotopic beams can be utilized either for target preparation or for studies of ion collision phenomena. For this last purpose the machine is also ideal, since it gives a monoenergetic, well focused ion beam.

3. Isotope separator in Gothenburg

The electromagnetic isotope separator in Gothenburg was completed in 1953 and was built for the production of targets for the electrostatic generator at the Physics Division and the eventual preparation of radioactive samples. Nowadays pure isotopic samples of practically all stable elements can be obtained from the large isotope production centres. However, it has proved to be a great advantage having an isotope separator at the same laboratory as the electrostatic generator. Very often the thickness of the target for the generator as well as the backing material must be changed after preliminary measurements have been performed. This means that many targets are necessary before an investigation can be completed. It is easy to understand that a lot of time and money is saved, with both machines available in one laboratory.

The main parameters of the machine were chosen as follows: A mean radius of 100 cm gives 4 mm between adjacent isotopes at mass number 250, when the target is placed at the radius distance from the exit boundary of the magnet — 90° sector machine. 50 keV maximal ion energy allows from some ten up to a few hundred μA ion current to be used at a line half width of 1–3 mm. This means that even the heaviest masses can be resolved fairly well. For further data see paper I.

In the following a few special problems will be treated:

4. Ion sources

4.1. Short note on ion source physics

Development work and systematic investigations on ion sources were made by K. O. Nielsen⁴), and O. Almén and K. O. Nielsen (paper II) in 1955—57. Since that time an ion source, originally built in Copenhagen, has been used in connection with the Gothenburg isotope separator. It consists of a magnet coil and a discharge chamber (fig. 2). The discharge chamber contains a filament, electrically connected to the plate behind it, an anode and an outlet plate, connected either to the filament or to the anode. At a certain, well defined gas pressure in the discharge chamber and with the necessary electrons emitted from the filament, a gas discharge is started and an ion — electron plasma created. From this plasma electrons and ions must leave with the same rate as they are produced. The ions go to the filament and to the outlet plate, where some of them pass the outlet opening. With an electric field outside the source, penetration of this field through the opening causes a curved plasma boundary, called the plasma cap. The electrons must go to the anode. With the outlet plate connected to the filament the electrons oscillate in the chamber before being lost to the anode; known as oscillating electrons. The electrons cannot go directly to the anode, because they are kept together into a quite narrow beam by the magnetic field, directed parallel with this electron beam. With the outlet plate connected to the anode most of the electrons strike the outlet plate after one passage through the discharge chamber; known as direct electrons. The higher electron consumption in this case is compensated by a higher filament heating current. This means a shorter lifetime of the filament and therefore oscillating electrons are mostly used. Gas is fed to the source from a container via a needle valve, or from a charge container, placed in a heated oven behind the discharge chamber.

4.2. Construction of discharge chambers

This ion source with ion extraction parallel with a magnetic field ("end extraction") has been very reliable and various types of discharge chambers have been built to accommodate the different requirements of the elements processed. It only takes a few minutes to replace

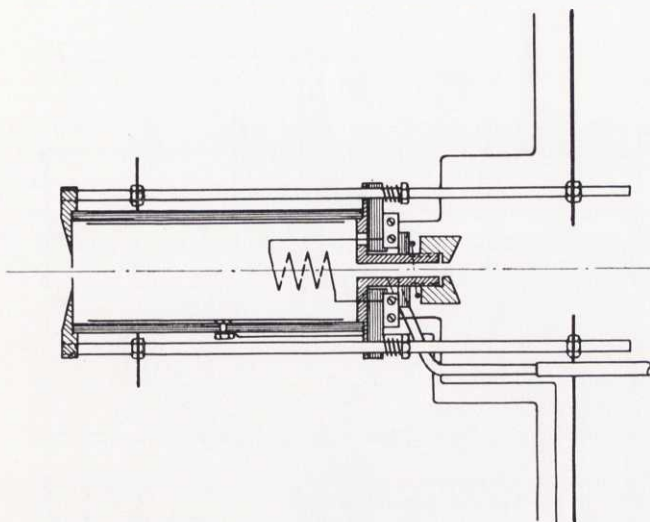


Fig. 3. Discharge chamber for gases. Quarts tube and stainless steel plates.

the discharge chamber with a new one of the same type or with one of another type. Sometimes it is necessary to change the discharge chamber between separations, since the different elements may interfere with each other. There is always a small memory in the source, because of the penetration of ions into the walls and sometimes due to condensation on a part of the source being too cold. This happens in particular when a chemical compound is used as charge material, the element itself having too low vapour pressure at temperatures conveniently obtainable in the discharge chamber. In the plasma the compound is broken up and material with low vapour pressure is deposited on the walls of the discharge chamber. Part of this material comes off through reaction with the other component of the compound, but part of it remains and can give a volatile compound with any of the components of another charge material used later on. Separation of radioactive material usually means a contamination of the source so that it may be necessary to allow a "cooling down" period before the discharge chamber can be used again.

Four different types of discharge chambers have been used in connection with the above mentioned magnet chamber.

- I: A simple, low temperature chamber used only for gases is shown in fig. 3. It is constructed of stainless steel and a ceramic or quartz tube. It is very easy to handle and can be operated up to one hundred hours without any trouble.

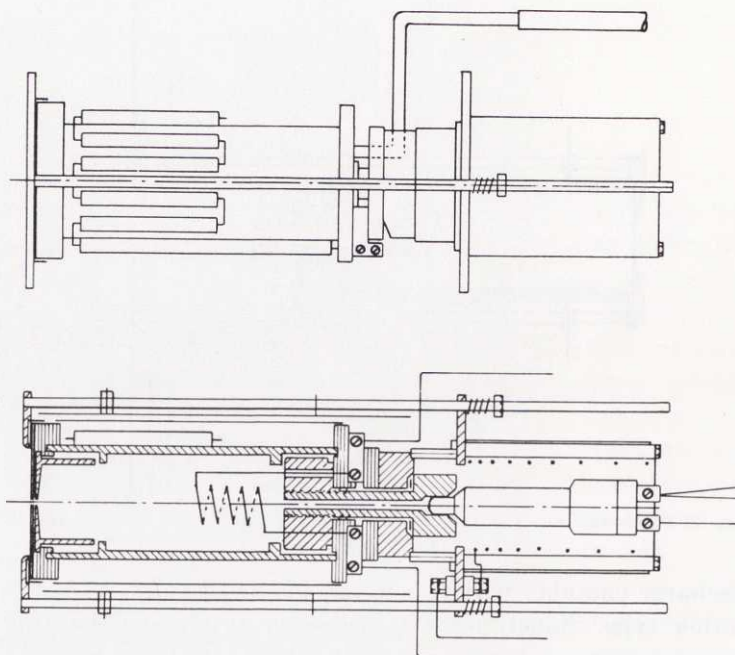


Fig. 4. Discharge chamber for medium temperatures.

- I b: The same as I but with a larger filament and the chamber surrounded by radiation shields in order to reach a temperature of $500-600^{\circ}\text{C}$. This has been used for calcium and strontium but failed for barium. These three elements cannot be handled in a discharge chamber made of graphite, which is used at high temperatures, because they all react easily with carbon forming carbides.
- II: A medium temperature graphite discharge chamber with insulators of pyrophyllite is shown in fig. 4. This has been used for most of the pure elements and for many compounds requiring medium temperatures of $500-800^{\circ}\text{C}$. The restriction of temperature is because pyrophyllite cannot stand the chemical attack of e.g. chlorine at high temperature.
- III: The high temperatures necessary for handling the rare earth elements required construction of a unit which at $800-1000^{\circ}\text{C}$ can stand a chlorine atmosphere, without giving off impurities (fig. 5). Quarts has proved to be a suitable material for insulators.

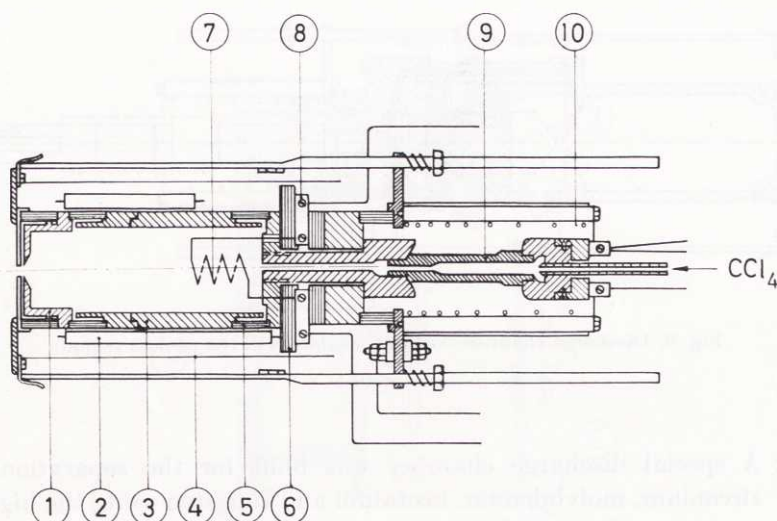


Fig. 5. Discharge chamber for high temperatures. (1) outlet plate, (2) heating elements of Mo surrounding the anode cylinder (3) of graphite, (4) shielding of Mo, (5) insulators of quartz, (6) insulator of BN, (7) filament, W, (8) connections to filament, Mo, (9) charge container, (10) oven of Mo wire.

To avoid sputtering of the insulator surfaces these are well shielded from the plasma. This discharge chamber can be used for direct electrons in which case more power is dissipated on the outlet plate giving a more smooth temperature distribution in the chamber. Anode heating and radiation shields are necessary but of course it can be operated without these at lower temperatures.

Fig. 5 also shows a chlorinating equipment used for the rare earth metals. The method was developed by G. Sidenius and O. Skilbreid⁵⁾ at Copenhagen, and means that CCl_4 is fed through quartzwool containing the oxide heated to about 800°C . Chloride is formed and evaporates into the discharge chamber. Very high efficiencies have been obtained at Copenhagen with this method, using mg quantities of charge material. Chlorinating has been applied successfully by us for a few other elements namely: beryllium, boron, aluminium, vanadium, germanium, tin and barium, but did not work for titanium and hafnium. G. Sidenius and O. Skilbreid have reported good results for the rare earths and also for hafnium and tungsten.

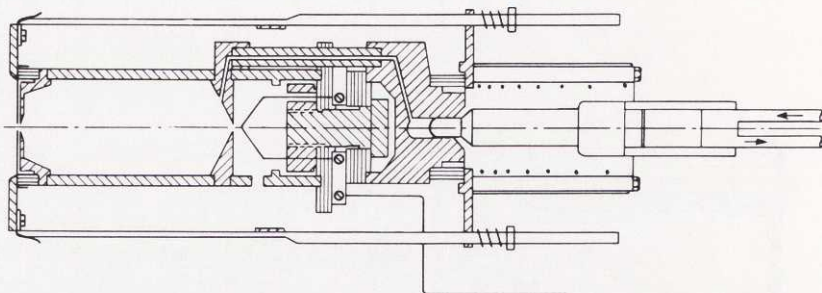


Fig. 6. Discharge chamber without insulators in the plasma region.

IV: A special discharge chamber was built for the separation of zirconium, molybdenum, tantalum and tungsten using the highly corrosive compounds with very high vapour pressures ZrCl_4 , MoCl_5 , TaCl_5 and WCl_6 (fig. 6). No insulators are contained in the discharge chamber which is made of graphite. Electrons are supplied to the plasma through a small opening. A water-cooled charge container is used and with this it is possible to reach temperatures as low as $30-40^\circ\text{C}$. The water-cooled container has also been used in connection with discharge chamber II for temperatures in the region of $100-200^\circ\text{C}$. Discharge chamber III can of course be used instead of II and can probably replace also IV. However, III was the last to be constructed so therefore II and IV were built. A description of all the different types we have used, seems worthwhile, however, because it reviews the difficulties in processing the different elements.

V: A few elements have a very low vapour pressure even at 1000°C and suitable chemical compounds do not exist. For these elements a sputtering discharge chamber has been used (fig. 7). The operation of the sputtering type source was originally described by J. Druaux and R. Bernas⁶). The discharge is operated on e.g. argon gas. An electrode of the material from which ions are wanted is fed with a negative voltage of $500-1000\text{ V}$. Argon ions are drawn from the plasma and sputter off atoms which are ionized in the same plasma. The efficiency of the arrangement is very poor, but it was possible to use it for our experiments on collection and sputtering, (paper IV) using ions of e.g. palladium, platinum and gold.

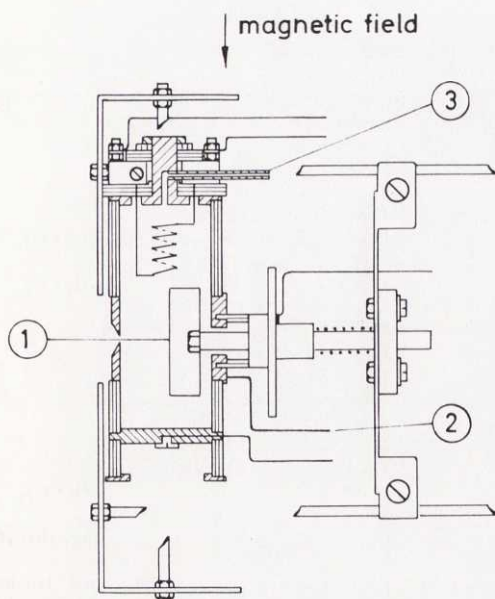


Fig. 7. Sputtering discharge chamber. (1) negative electrode, (2) anode, (3) gas inlet.

Discharge chamber V was operated in an ion source with the magnetic field perpendicular to the direction of the ion beam. Several experiments using the end extraction type of ion source with different positions of the high tension electrode all failed to give sufficient current.

4.3. Charge material

In table 1 are listed charge materials, ion source types, I—V, oven temperatures and additional remarks for all elements up to bismuth, most of which have been processed at Gothenburg. In a few cases more than one choice is given, sometimes the temperature is extrapolated by means of a vapour pressure chart using data reported by the Oak Ridge National Laboratory⁷).

Normal vapour pressures in the discharge chamber are of the order of 10^{-3} mm Hg. If a charge material has a vapour pressure of a few mm Hg at room temperature, it can be fed to the chamber via a needle valve. Corrosion sometimes causes trouble, e.g. using SiCl_4 and TiCl_4 the needle valve can be blocked up. For higher temperatures a charge container in a heated oven is used. Below 300°C temperature

TABLE 1

Element	Charge material	Type of discharge chamber	Oven temp °C	Remarks
He	He	I		
Li	LiCl	II	500	
	Li a)		~ 400 b)	
Be	Be	II	550—650	with CCl ₄
	BeCl ₂ a)		~ 150 b)	
B	B	II		with CCl ₄
	BCl ₃ a)		~ 150 b)	
C	CO ₂	II		
N	N ₂	I		
O	O ₂	I		
F	—	—		
Ne	Ne	I		
Na	NaCl	II	420—440	
Mg	Mg	Ib, II	310—350	
Al	Al	II		with CCl ₄
Si	SiCl ₄	IV		
P	P ₂ O ₅	II	200	long, throttled tube
S	CS ₂	II		
	S	II	~ 100	long, throttled tube (reacts with stainless steel)
Cl	CCl ₄	II		
Ar	Ar	I		
K	KBr	II	410—425	
	K a)		~ 100 b)	
Ca	Ca	Ib	420—500	stainless steel only
Sc	SeCl ₃	II, III	400—500	
	Se-oxid	III	700	with CCl ₄
Ti	TiCl ₄	V, III		through needle valve
V	V-oxides	III		with CCl ₄
Cr	CrCl ₃	II	420—425	
Mn	MnCl ₂	II	350—400	
Fe	FeCl ₂	II, III	310—360	long throttled tube
Co	CoCl ₂	II	400	
Ni	NiCl ₂	II, III	380—420	long throttled tube
Cu	CuCl	II, III	260—280	long throttled tube
Zn	Zn	I, II	130	water cooled
Ga	GaI ₃	III	60	long throttled tube, water cooled
Ge	Ge	II	400	with CCl ₄
	GeCl ₄ a)			
As	As	I	90	water cooled
Se	SeO ₂	I	60—70	water cooled
Br	KBr	II	410—425	
Kr	Kr	I		
Rb	RbCl	Ib, II	350—450	
Sr	Sr	Ib	480	stainless steel only
Y	YCl ₃	II	540—600	
Zr	ZrCl ₄	IV	130—150	long throttled tube
Nb	Nb	V		
Mo	MoCl ₅	IV	50—60	long throttled tube, water cooled
	Mo	V		
Tc	—	—	—	no stable isotope

Element	Charge material	Type of discharge chamber	Oven temp C	Remarks
Ru	—	—	—	CCl ₄ does not work sputtering ion source should be possible
Rh	—	—	—	CCl ₄ does not work
Pd	Pd	V	—	
Ag	AgCl	II, III	550—590	
Cd	Cd	I, II	250 60—70	long throttled tube water cooled
In	InI	III	110	long throttled tube, water cooled
Sn	Sn SnCl ₂	II II, III	500 120—130	with CCl ₄ water cooled
Sb	Sb	II	240—300	long throttled tube
Te	Te	II	300—310	long throttled tube
I	I	II	—	through needle valve
Xe	Xe	I	—	
Cs	CsCl	II	400—410	
Ba	BaO Ba a)	III	600—700 ~ 600 b)	with CCl ₄ stainless steel only
La	La La ₂ O ₃	III	820	with CCl ₄ with CCl ₄
Ce	CeO ₂ CeCl ₃	III III	550	with CCl ₄
Pr	PrO ₂	III	700	with CCl ₄
Nd	Nd ₂ O ₃	III	740	with CCl ₄
Pm	—	—	—	no stable isotope
Sm	Sm ₂ O ₃	III	800	with CCl ₄
Eu	Eu ₂ O ₃	III	800—850	with CCl ₄
Gd	Gd ₂ O ₃	III	820	with CCl ₄
Tb	Tb ₂ O ₃	—	—	with CCl ₄ c)
Dy	Dy ₂ O ₃	III	800	with CCl ₄
Ho	Ho-oxides	—	—	with CCl ₄ c)
Er	Er ₂ O ₃	III	~ 800	with CCl ₄
Tm	Tm ₂ O ₃	—	—	with CCl ₄ c)
Yb	Yb ₂ O ₃	III	~ 800	with CCl ₄
Lu	—	—	—	CCl ₄ on oxide should work
Hf	HfCl ₄	III	80	long throttled tube, water cooled
Ta	TaCl ₅	IV	30—40	long throttled tube, water cooled
W	WCl ₆	IV	80	water cooled
Re	Re ₂ O ₇ a)	—	~ 150 b)	
Os	—	—	—	
Ir	—	—	—	sputtering source should be possible
Pt	Pt	V	—	
Au	Au	V	—	
Hg	HgO	II	120—130	water cooled
Tl	Tl	II	450	
Pb	Pb	II	530	
Bi	Bi	II	480	

a) Oak Ridge National Laboratory⁷⁾

b) Estimated from vapour pressure chart

c) Sidenius, Skilbreid⁸⁾

regulation of the oven is very poor, since only a small part of the heat is lost by radiation and conduction losses work slowly. Furthermore some heat is conducted back from the discharge and may raise the oven temperature too much. The best arrangement is to use a water-cooled charge container. However, if a long thin canal is used between the charge container and the discharge chamber the flow resistance is increased and the temperature can be kept much higher and thus be under control. Normal tube length is ~ 50 mm, a long tube means 80 mm with inside diameter 2 mm. This can be throttled with a piece of graphite or stainless steel of e.g. 1.8 mm diameter, giving the required low conductance.

5. Ion beam formation

In this chapter the following symbols are used:

- A_0 = area of the outlet opening
- A_p = area of the plasma cap
- $B_{\frac{1}{2}}$ = line half width at the collector
- d = distance between the ion source and the extraction electrode
- I_i = ion current measured at the collector
- I_i^m = ion current measured at the collector at optimal focusing in the "1:st state".
- I_s = electron current going from the extraction electrode to the ion source
- K = ion production rate in the discharge chamber
- M = mass number
- p = pressure in the discharge chamber
- Q = effective space charge in the acceleration gap
- V_a = arc voltage
- V_h = acceleration voltage
- V_l = lens potential
- \varnothing = diameter of the outlet opening
- \varnothing_I = diameter of the opening in the extraction electrode
- η_{\max} = maximal total efficiency — from gas inlet to collector

5.1. Discussion

Although it is necessary to use an ion source suitably constructed to obtain a high ion current and an ion current of only one element, the source itself does not put a limit to the number of μA which can

be used at the collector. Normally the source can produce much more current than can be utilized by the beam handling device — the lens system and the deflection magnet. In order to get the best focusing at the target end of the isotope separator, that is the highest resolving power of the instrument, the ion beam must have a fixed divergence when entering the magnetic deflection field. This beam divergence is determined by the following sets of parameters:

- a) parameters inside the ion source: filament heating current, arc voltage, magnetic field and gas pressure in the discharge chamber. All these influence the ion density in the plasma discharge which on the other hand determines the shape of the plasma boundary at the outlet.
- b) parameters in the acceleration gap: acceleration voltage, distance between the ion source and the acceleration electrode, outlet area, shape of electrodes, and effective space charge density in the acceleration gap. Since the ion beam has its smallest cross section just outside the source, the space charge spread is here more pronounced than along the rest of the beam, and the beam divergence is determined already here. The space charge spread must depend on the effective space charge density which in general is composed of an ion current leaving the ion source and an electron current returning to the source.
- c) the principle parameter of the electrostatic lens system: lens potential.

The ion production rate, K , in the plasma is determined by the parameters inside the source (listed under a) above). A discussion of the ion production rate and the influence on it of the different parameters is found in paper II. K , is however, most sensitive to variations of the filament heating current, that is the electron input to the plasma. With an electric field applied between the source and the first electrode of the lens system ions can be drawn out from the plasma through the outlet opening. Because of the penetration of the electric field the plasma boundary is pushed back into the source and adjusts itself to a position where there is balance between the number of ions reaching the boundary from the production region and the number streaming out into the acceleration gap. The ion current, I_i , reaching the collector of the isotope separator is determined by the parameters listed under b) above.

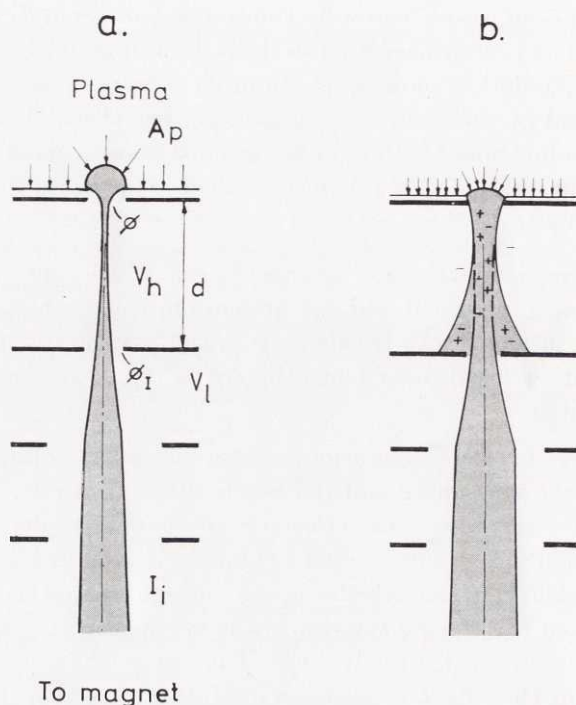


Fig. 8. Shape of the plasma boundary and the extracted ion beam in a) "1st state", b) "2nd state" of operation.

We will now discuss variations of I_i and $B_{\frac{1}{2}}$, the line half width at the collector, when the ion production rate, K , is increased by increasing the filament heating current and keeping the rest of the parameters a) constant. All parameters b) except the space charge density are also kept constant. The lens potential is adjusted for best focusing.

When K is low the ion emitting plasma cap, with area A_p , bulges deep into the discharge chamber. Since the boundary is an equipotential surface the ions leave the cap perpendicularly and converge into a narrow ion beam, fig. 8 a. By adjusting the lens potential, V_l , the beam divergence at the entrance of the magnetic field can be given a proper value, so that the isotopic beams reaching the collector have a line half value, $B_{\frac{1}{2}}$, of the order of 1 millimetre which means that they are well resolved. The space charge density, Q , in the beam is so low that the focusing obtained by the concave plasma cap compensates the space charge spread. Also a few electrons, produced by

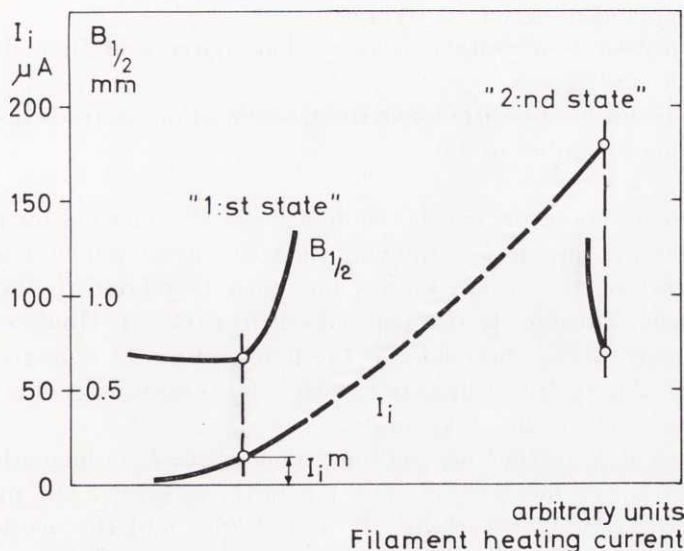


Fig. 9. Ion current and line half width vs. filament heating current. $\varnothing = 2.0$ mm, $d = 55$ mm and $V_a = 75$ V.

collisions in the rest gas, decrease Q . However, very few electrons are produced at a rest gas pressure of $5 \times 10^{-6} - 10^{-5}$ mm Hg at such a low Q value. Increasing the rest gas pressure has always worsened the focusing in our machine. In large machines increasing the pressure is a method to obtain better focusing.

With increasing K , I_i increases and $B_{\frac{1}{2}}$ remains practically constant up to a certain value, above which $B_{\frac{1}{2}}$ suddenly increases very fast (fig. 9). At this point, which is quite well defined, the plasma cap and lens potential focusing still manage to give a beam which can be focused at the collector. It was used as a criterion for the systematic investigation described in paper II. In this it was experimentally shown that the ion current I_i^m obtained under this very special condition approximately follows the formula for a space charge limited current flowing between plane electrodes:

$$I_i^m = C(\theta) \frac{A_0}{d^2} \frac{V_h^{3/2}}{M^{\frac{1}{2}}} \quad (1)$$

$C(\theta)$ = a constant depending on the shape of the electrodes constituting the acceleration gap;

- A_0 = area of outlet, 3.5—20 mm²;
 V_h = acceleration voltage between ion source and first electrode
 5—70 kV;
 d = distance between ion source and acceleration electrode 15-70mm;
 M = mass number of ion.

Of course objections can be made against this method for making investigations on an ion source, since the measurements are not representative for the ion source, but must be related to the whole instrument. This fact is also pointed out in paper II. However, a lot of information was obtained and the practical use of a magnetic ion source of this type in connection with a 90° electromagnetic isotope separator was very much simplified.

Increasing K further on gives a region where I_i is increasing and $B_{\frac{1}{2}}$ is no longer measurable. The ion beam is spread out over the whole collector. At a certain adjustment (fig. 8 b) the acceleration electrode is hit by a part of the ion beam. Every ion impinging on the electrode produces a number of secondary electrons which because of the electric field go to the outlet plate of the ion source. Strange effects appear sometimes probably due to a misalignment of the source and the lens system. The returning electron beam can, e.g. be focused on a spot a small distance from the outlet opening accompanied with an enormous heating up of the spot. Materials such as molybdenum and stainless steel can be melted easily when such spots are formed. Normally the electrons impinge on a larger area and no trouble is caused.

Some of the electrons travel into the ion beam on their way to the ion source and decrease the positive space charge density within the ion beam in the acceleration gap. Thus the heavy positive space charge is partially compensated for and the space charge spread is counteracted. When enough electrons are produced and brought into the ion beam we reach a situation where we get a dense central ion beam containing a lot of electrons for space charge compensation, surrounded by a beam for production of the necessary electrons through impact on the acceleration electrode. The central beam has such properties that it can be handled by the lens system and the magnet giving very well focused isotopic beams at the collector. In fact, the resolving power of the instrument is the same or even higher using this beam than using the low density beam mentioned above. This can be seen from fig. 10 b which will be discussed below.

In paper II the two states of operation have been mentioned. The "first state" received most of our interest in this paper and could be fairly well investigated and understood. The "second state" was explained by the assumption that the plasma penetrated through the outlet opening into the acceleration gap and there gave about the same ion emitting conditions as in the "first state". Although this explanation was not proved and not at all understood, it seemed to be quite probable. However, the investigations made here do not require the plasma to penetrate the outlet opening. Instead the beam shape indicates a continuous process up to the point where electrons start returning to the source, without an abrupt change which would be likely due to a shift of the boundary from inside to outside the source.

That the beam formation process really is that indicated above has been checked by applying an acceleration electrode containing a filament for electron supply to the ion beam by other means than by ion impact on the electrode. Approximately the same ion current and line half width can be obtained with and without the extra electron supply. This is strong evidence that the presence of the electrons develops the condition for the "second state". The filament heating current in the ion source must be decreased, when electrons are supplied from the filament in the acceleration electrode, indicating that it is no longer necessary for the beam to hit the electrode.

If the ion production rate or the returning electron current is increased above that corresponding to the "second state" focusing is no longer possible. Instead the electron current to the source increases very fast and overloads the high voltage supply.

The electrons going to the ion source, connected to high tension 45 kV, produce of course x-radiation, which can be very intense, e.g., 25 mr/h has been measured at 1 metre distance from the ion source.

5.2. Measurements of ion current and line half width

Although many problems about beam formation are not understood at the moment a few of the measurements made with our instrument will be presented in the following. Only krypton ions have been used for the investigations and the measurements have been limited to practical questions such as, what ion current and total efficiency can be obtained using the "second state" of operation rather than the "first state". A discharge chamber of type III (fig. 5) of length 70 mm

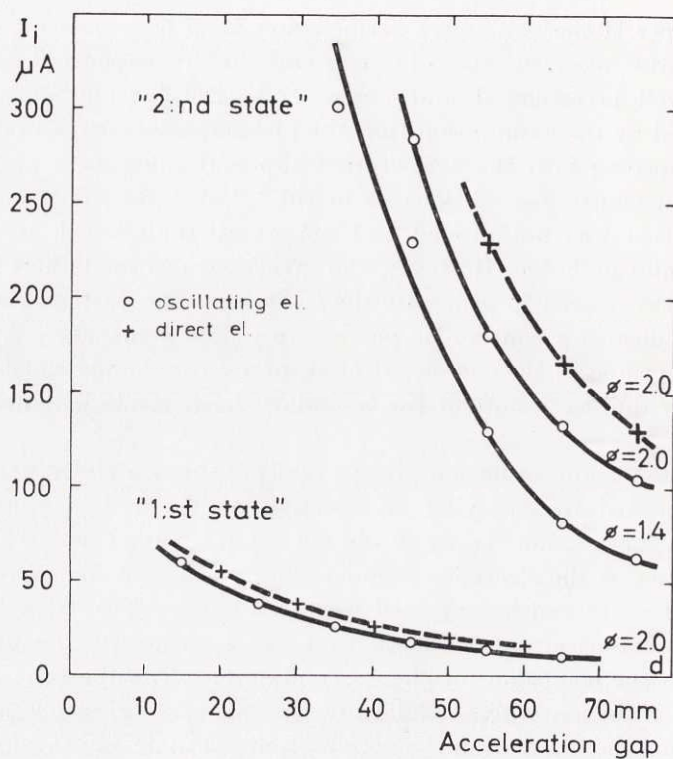


Fig. 10 a. Ion current vs. distance d between ion source and extraction electrode for different outlet diameters. $V_a = 75$ V.

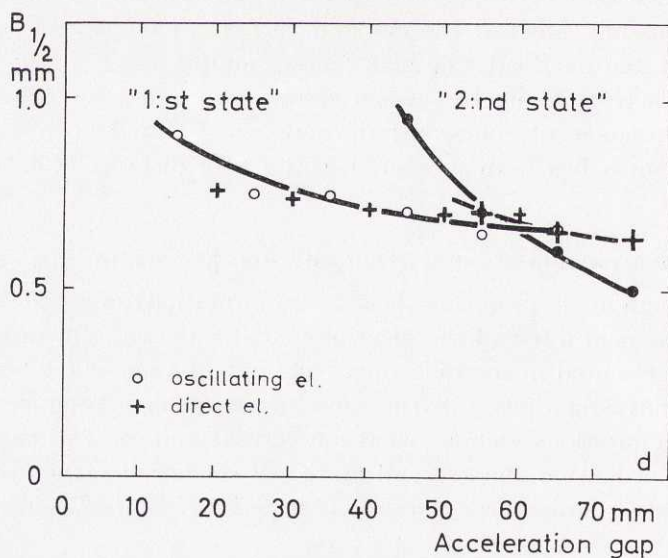


Fig. 10 b. Corresponding line half widths $\phi = 2.0$ mm, $V_a = 75$ V.

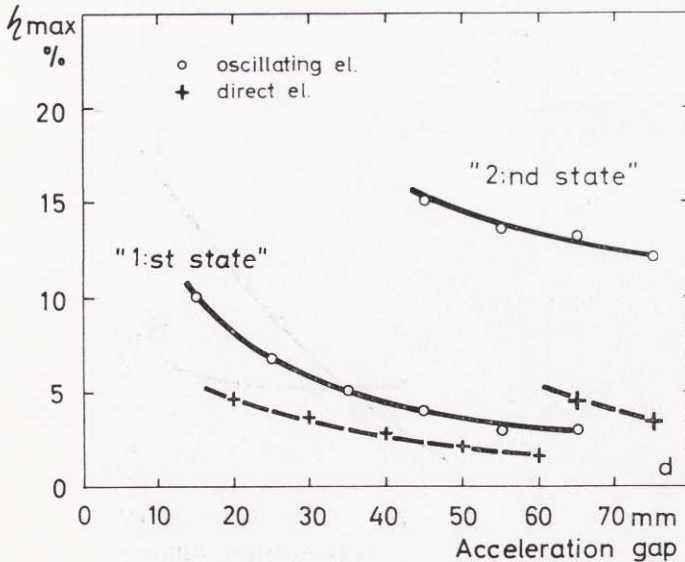


Fig. 11. Maximal total efficiency vs. distance d . $\phi = 2.0$ mm, $V_a = 75$ V, length of the discharge chamber 70 mm.

has been used in all measurements described here. If nothing else is pointed out we have used in all measurements acceleration voltage 45 kV, opening diameter of the extraction electrode $\phi_I = 9$ mm made of stainless steel and at ground potential, and the magnetic field in the ion source constant, 100 gauss.

Fig. 10 a shows the ion current measured at the collector for the two states using both oscillating and direct electrons in the ion source. From fig. 10 a we see that there is approximately a factor of 10 between the currents in the two states, while as indicated in fig. 10 b the line half widths, $B_{\frac{1}{2}}$, are approximately the same. $B_{\frac{1}{2}}$ has been measured on the oscillograph screen, where the output from the mechanical scanner is displayed (described in paper I). The linear amplification from the collector to the oscillograph is about 4.5 times, so line widths less than 1 millimetre can be easily measured. With direct electrons a few per cent more current is obtained than with oscillating electrons. Probably the different position of the plasma cap causes the small difference.

A comparison between measurements for outlet openings $\phi = 1.4$ and $\phi = 2.0$ mm indicates that eq. 1 will not hold for a variation of the outlet in the "second state", although the $1/d^2$ variation is

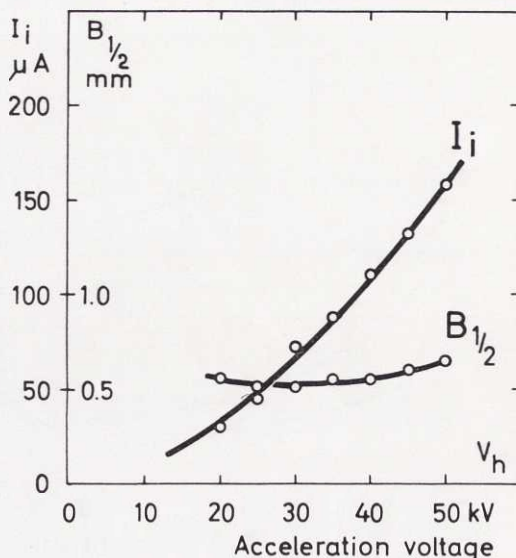


Fig. 12. Ion current and line half width vs. acceleration voltage.

$\varnothing = 1.4$ mm, $V_a = 75$ V, $d = 55$ mm.

approximately fulfilled as well as the $V_h^{3/2}$ variation (fig. 12). All discussions concerning a formula for I_i in the "second state" must, however, be abandoned after taking into account the results presented in figs. 13 and 14. A variation of arc voltage, V_a , and krypton pressure, p , in the discharge chamber changes both I_i and $B_{1/2}$ considerably for the "second state", while "first state" data are quite insensitive to both parameters. Operating at the minimum pressure, however, decreases the differences and all curves go to approximately the same value. The variations with V_a and p are caused by the different ways of producing the necessary electrons for space charge compensation. On the other hand the magnetic field in the ion source, does not influence I_i and $B_{1/2}$ in either state.

Electron production and the accompanied space charge compensation can be obtained under different conditions as shown clearly in fig. 14. Both the diameter of the opening in the extraction electrode, \varnothing_I , and the electrode material influence the ion current in the "second state". The first determines at what ion production rate secondary electrons start to influence the ion beam. Different materials have different coefficients for secondary electron emission. From the explanation of the conditions for the "second state" above, it is there-

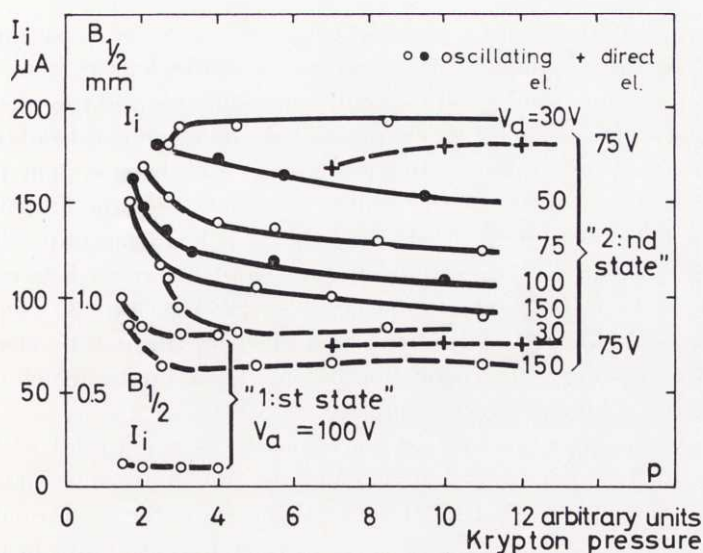


Fig. 13. Ion current and line half width vs. pressure in the discharge chamber with arc voltage as parameter. $\varnothing = 2.0$ mm, $d = 65$ mm.

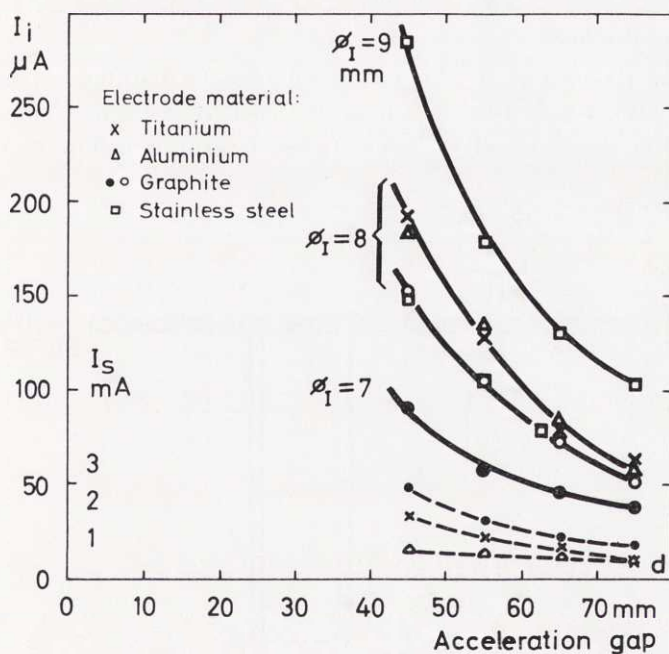


Fig. 14. Ion current vs. distance d , with opening diameter \varnothing_I and material of extraction electrode as parameters. $\varnothing = 2.0$ mm, $V_a = 100$ V. Electron current I_s from the extraction electrode is also shown.

fore obvious that \varnothing_1 and a material constant will enter as parameters, as well as the alignment of the source and the lens system. Thus measurements of I_i and $B_{\frac{1}{2}}$ are not fully reproducible and the presented results must be regarded as examples only of what can be obtained.

We now always operate the ion source and lens system in the "second state", because of the gained factor of 10 of the ion current, which sometimes is very valuable. Hitherto it has been impossible to use larger outlet diameters than 2.5 mm, and distances between the ion source and the extraction electrode smaller than 35–45 mm. The reason is that the ion beam starts hitting the middle electrode of the lens system, when operating outside these limits, which causes all focusing to break down completely.

The extraction electrode should be made of a material with low coefficients for secondary electron emission and sputtering. Titanium, nickel and stainless steel seem to be the best materials. Aluminium has been tried, but it is eroded away by sputtering quite fast.

The amount of doubly charged krypton ions and ions from foreign materials has been about 5 per cent of the measured ion current.

5.3. Total efficiency

In order to complete the picture of the two different states of operation the total efficiency has also been measured. Using an oil manometer, schematically shown in fig. 15, the gas flow to the ion

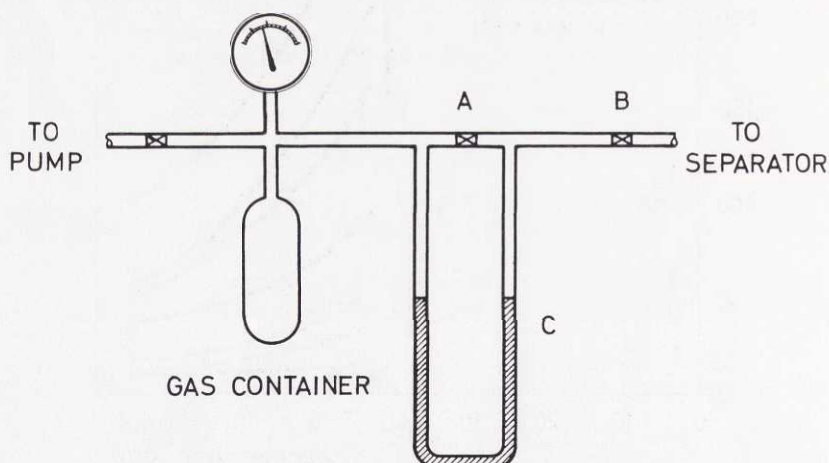


Fig. 15. Arrangement for determining gas quantities. 1) valve A open. 2) adjust B to minimum pressure. 3) close A and measure time for a suitable pressure change indicated by C.

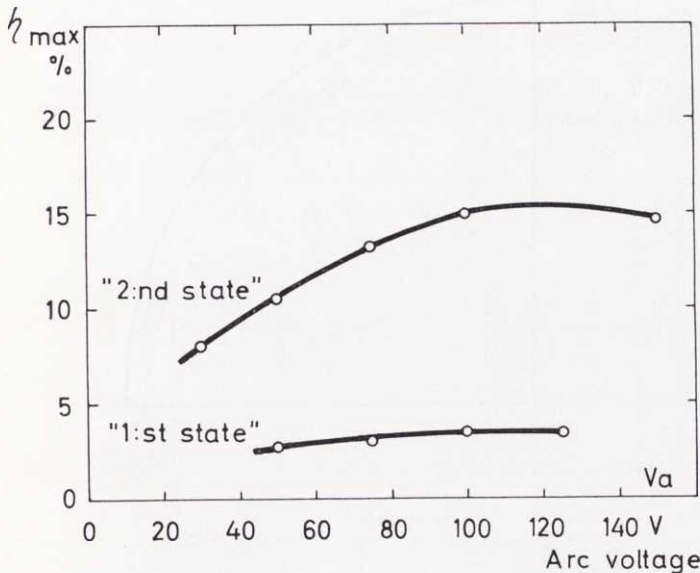


Fig. 16. Maximal total efficiency vs. arc voltage. $\varnothing = 2.0$ mm, $d = 65$ mm, length of discharge chamber 70 mm.

source can be determined. If this is done immediately after adjusting to minimum pressure in the discharge chamber and measuring the ion current reaching the collector the efficiency can be calculated. Figs. 11 and 16 show η_{\max} for the two states measured under equal conditions. η_{\max} is approximately 4 times higher for oscillating electrons in the "second state" than η_{\max} for the "first state". Direct electrons give in both states lower efficiency than oscillating electrons. This result is remarkable and there is no explanation hitherto.

6. Collection technique

Already in 1947 J. Koch et al.⁸⁾ collected small quantities of noble gases for nuclear research. The direct collection method was used, which means that an ion beam hits a metal plate and some of the ions are collected due to their penetration into the metal lattice. Only a few more investigations on this type of collection technique^{9 10)} were made until 1958, when experiments were started in Gothenburg in order to find how large quantities of noble gases could be collected in

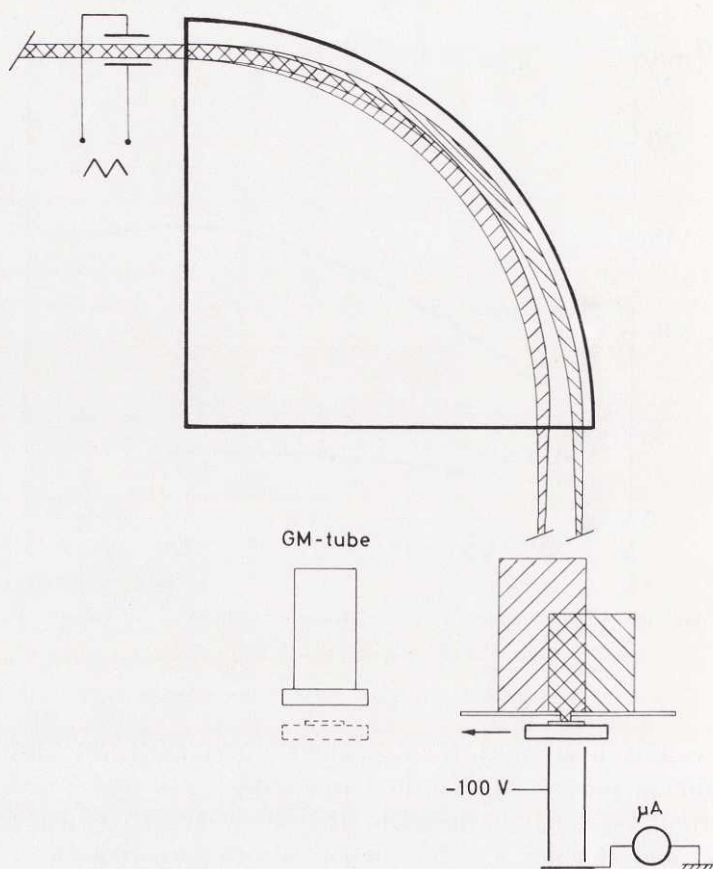


Fig. 17. Collector arrangement for saturation studies by means of the radioactive tracer method. A radioactive isotope is mixed with a stable by means of a linear sweep.

different backing materials and what the limitations were. This work is described in paper III.

The direct collection method is most convenient to use in a small laboratory machine. It means simple preparation directly in the isotope separator, and a high chemical and isotopic purity can be obtained. Only small quantities, $\mu\text{g}/\text{cm}^2$, can be handled. The purpose of our machine is to produce thin samples, as mentioned above, and for this work the obtainable quantities are in most cases sufficient. In large machines collection takes place in pockets, and a chemical process is necessary to get at the isotopes and make them available for the preparation of samples by means of evaporation technique or electrolytic deposition.

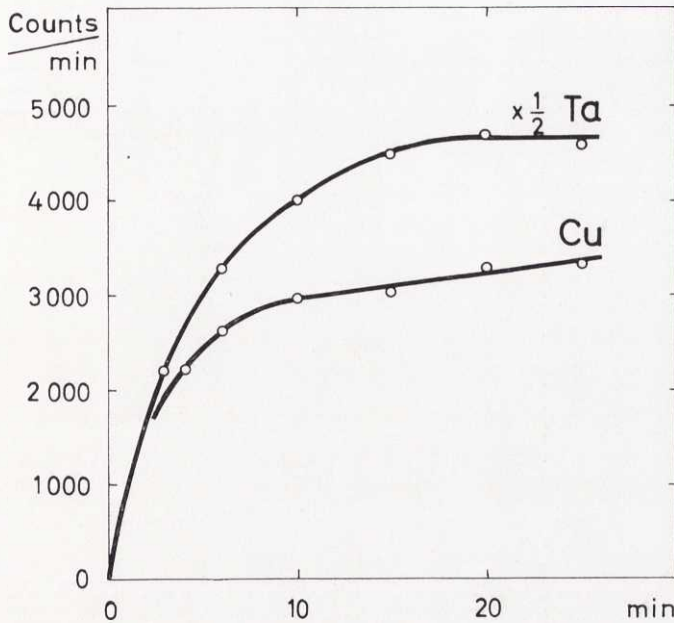


Fig. 18. Saturation curves for krypton collected in copper and tantalum at a current density $10 \mu\text{A}/\text{cm}^2$.

In the following we will distinguish between collection at high ion energy (30–50 keV) and collection at an energy below 5 keV, obtained after retardation of the high energy beam.

6.1. Collection at high ion energy

Radioactive tracer technique has been used to follow the increase of krypton and xenon amounts in different materials under ion bombardment. The used arrangement is schematically shown in fig. 17. A radioactive isotope is mixed with a stable one by means of a linear sweep voltage applied on plates at the entrance of the magnet. At the collector the activity increase is followed with a GM tube and a scaler. Typical collection curves are shown in fig. 18. At 45 keV ion energy the collected quantities are of the order of $1\text{--}5 \mu\text{g}/\text{cm}^2$ of krypton and xenon in different materials. The penetration depth is of the order of $20\text{--}50 \mu\text{g}/\text{cm}^2$, also varying with the target material.

Principally there is no difference between the collection of ions from a gas or a solid substance. The factors limiting the ion collection are in both cases a diffusion process and removal of target atoms (sputtering), because of the impinging high energy ions.

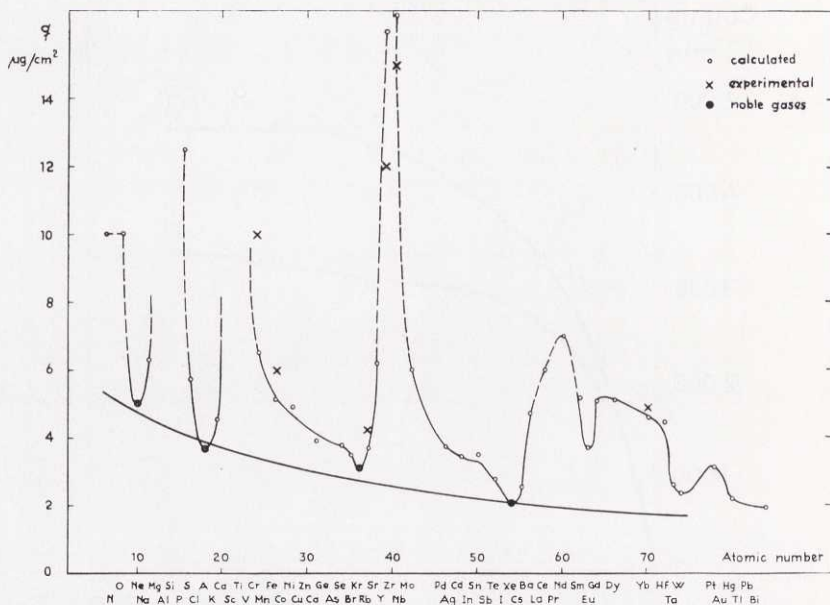


Fig. 19. Calculated and experimental saturation values for 45 keV ions bombarding a tantalum backing material.

With a few assumptions it was possible in paper IV to present the following formula, valid at keV ion energies, for the quantity q of ion mass M_1 , which can be collected at saturation in a backing material of atomic mass M_2 :

$$q = Cd_{12} \cdot \frac{M_1}{M_2} \cdot \frac{(1-r-D_i) - D_f}{(1-r-D_i) - D_f + S} \quad \mu\text{g}/\text{cm}^2 \quad (2)$$

Here it is assumed that q/M_1 is small compared to d_{12}/M_2 . where

C = a constant of the order of 2;

d_{12} = mean penetration depth of M_1 into M_2 in $\mu\text{g}/\text{cm}^2$;

D_f = fraction of impinging ions which leaves due to diffusion at saturation. D_f is a function of temperature and concentration of collected atoms;

D_i = fraction leaving due to diffusion, independent of the number of already collected particles. D_i is temperature dependent;

r = reflected ions in ions/ion;

S = sputtering ratio in atoms/ion.

$1-r-D_i$ is called the initial collection efficiency η_i , in paper III.

η_i is the probability for an impinging ion to be trapped in the metal lattice, and it is independent of the amount of atoms already collected. r is small compared to 1 and can be neglected in most cases.

By measuring q and S for the noble gases neon, argon krypton and xenon, q can be calculated by means of eq. 2 for a number of different kinds of ions collected in a 0.1 mm thick tantalum foil as backing material. (See paper IV). The result is given in fig. 19 for 45 keV ions. A few experimental results fit the calculated curve quite well. From this diagram it is easy to determine the time of collection for reaching saturation at a given current density (of the order of 10–100 $\mu\text{A}/\text{cm}^2$). It is also possible to answer the question, whether a given quantity required for an experiment can be collected or not using high energy ions. For the practical use of fig. 19, q can be assumed to vary about linearly with ion energy between 10–50 keV, if q is below $\sim 6 \mu\text{g}/\text{cm}^2$. Using other target materials the diagram must be modified. However, backing materials such as rolled, commercial foils of titanium, vanadium, iron, cobalt, nickel, zirconium, niobium, molybdenum, rhodium, tantalum and tungsten allow approximately the same amounts to be collected. Variations will not be greater than ± 30 per cent among these metals. In carbon and aluminium somewhat larger amounts can be collected.

The factors limiting the ion collection are as mentioned before, both sputtering and diffusion. Sputtering measurements have been discussed in considerable detail in papers III p. 268 and IV. In order to investigate the diffusion we have calculated D_f using eq. 2 for krypton ions of 45 keV bombarding different materials for which q , S and the initial collection efficiency η_i are experimentally known. η_i is determined by the slope of the first part of a collection curve such as given in fig. 18. In fig. 20 D_f is plotted as a function of the atomic number of the target atoms. For comparison η_i is also plotted in fig. 21 as a function of the atomic number at two different current densities, 10 $\mu\text{A}/\text{cm}^2$ and $\sim 0 \mu\text{A}/\text{cm}^2$. The last is obtained by using only the radioactive (krypton-85, beta active) beam without mixing it with a beam of a stable isotope. η_i at $\sim 0 \mu\text{A}/\text{cm}^2$ is given relative to that of aluminium, in which material η_i is expected to be close to one. The dependence of current density on η_i shows that D_i varies with the surface temperature of the bombarded metal. This result is also expected. In order to get comparable values the activity measurements must be corrected for different backscattering of the beta particles in

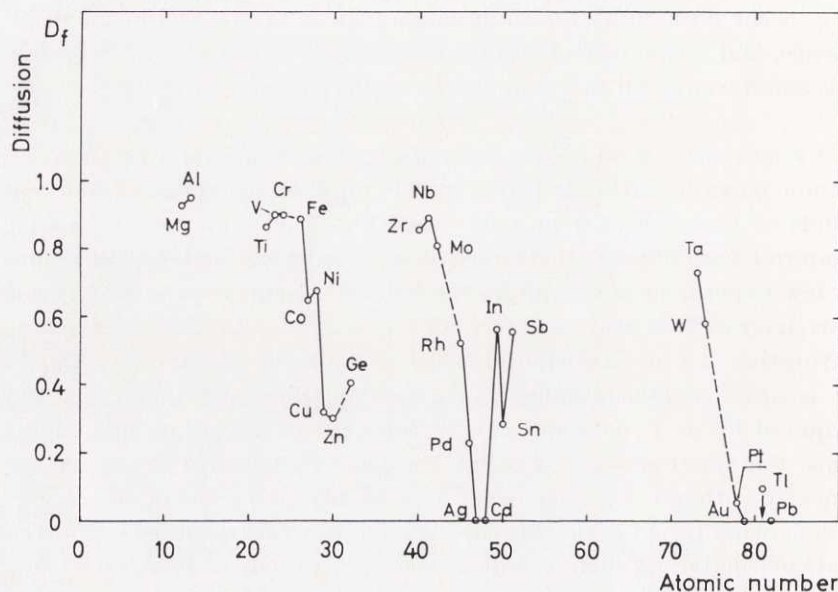


Fig. 20. Calculated diffusion at saturation for 45 keV krypton ions collected in different materials, current density $10 \mu\text{A}/\text{cm}^2$.

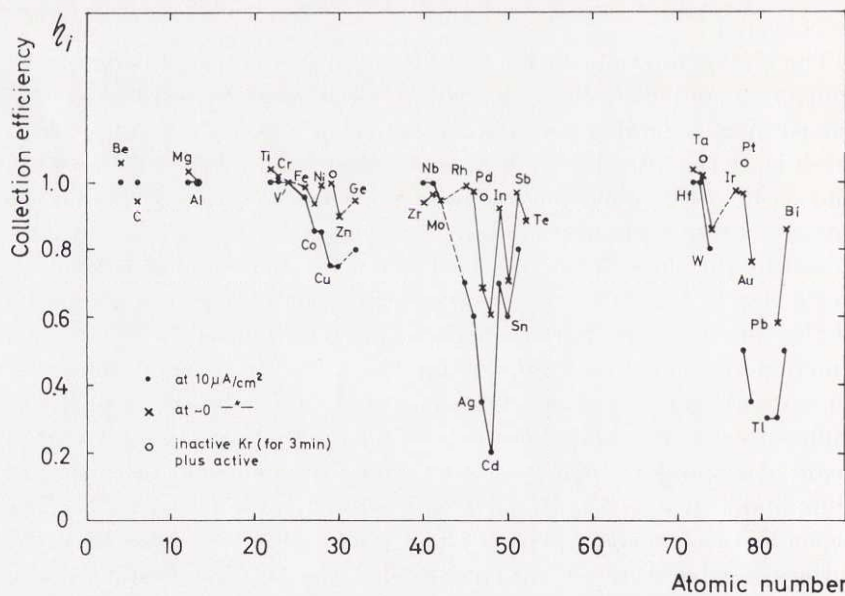


Fig. 21. Initial collection efficiency for 45 keV krypton ions. Current density ~ 0 and $10 \mu\text{A}/\text{cm}^2$, experimental error 0.05 and 0.1 respectively.

different materials. The correction factor has been determined experimentally.

A systematic variation of both D_i and η_i is obtained within the different groups of elements in the periodic system. From figs. 20 and 21 we see that, when $\eta_i \approx 1$, that is $D_i \approx 0$, D_f is also close to one. The sputtering ratio S is low for these elements. In order to get balance between impinging and leaving krypton atoms at saturation the only possibility is therefore a high diffusion D_f . Where there is a significant difference between the two plots in fig. 21, D_i is different from zero, and D_f must be below one. Here impinging atoms can leave as a result of two types of diffusion and the sputtering process. Where D_f is zero in fig. 20, D_i and S are large. Here so many of the η_i collected atoms are removed together with the sputtered S atoms that the concentration of krypton atoms is below the limit, where the diffusion D_f starts.

Another effect is also indicated in fig. 21. If the backing material is first bombarded with inactive krypton for a short time (3 min, 50 $\mu\text{A}/\text{cm}^2$) and then with active, a higher relative value for η_i is measured, than if only the radioactive beam is used. The collection in aluminium is not influenced by this procedure, while materials with a high sputtering ratio show a large change. The result can be explained. Sputtering removes the surface layer down to single crystal surfaces having a low sputtering ratio and correspondingly higher ability to store penetrating ions. It has been found in measurements using single crystals that a low sputtering ratio corresponds to a high saturation value and the reverse (see paper III p. 276). It is also expected that η_i will vary with the crystal structure. The mentioned effect of the sputtering can also be observed in some collection curves. At saturation there is still a small increase in some materials, while in others saturation means a practically constant value, see fig. 18.

This last observation indicates that collection of ions in a rolled material is a very complicated phenomenon, depending on parameters of the material not under control. Instead all studies of collection, diffusion and also sputtering should be performed in single crystals of known orientation. It is obvious that this means a laborious task, since many different crystal orientations must be investigated.

Fig. 22 shows a few sputtering measurements performed under exactly the same conditions for three different qualities of copper. The differences are therefore more accurate than the absolute values. Also here a dependence on material structure is seen which is explained

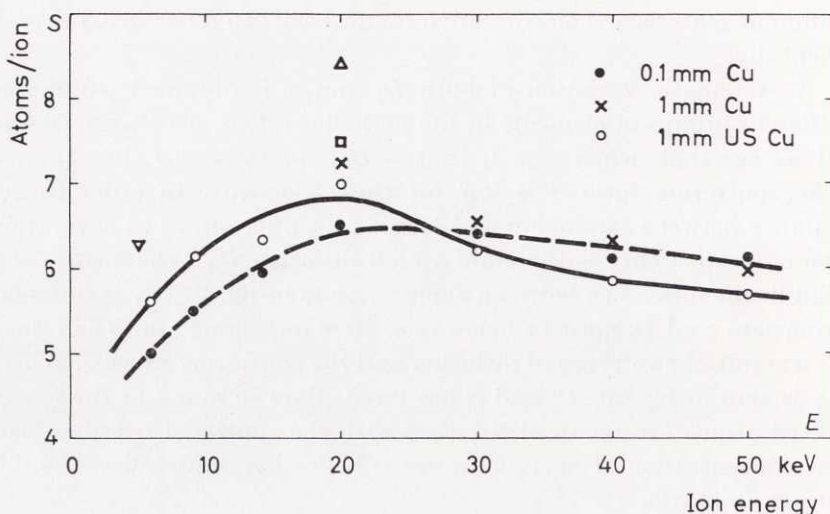


Fig. 22. Sputtering ratio of argon ions on different qualities of copper. Vacuum $3 \cdot 10^{-6}$ mm Hg, current density 1 mA/cm², experimental accuracy ± 5 per cent. Measurements on copper, 1 mm sheet from USA, made by other groups are indicated: Δ O. C. Yonts Oak Ridge, \square J. Kistemaker Amsterdam, ∇ M. T. Robinson Oak Ridge. Private communication with M. T. Robinson.

by the presence of preferred crystal orientations, even in a rolled material, said to be polycrystalline. Thus all values presented here must be regarded as examples of what may be obtained.

Collection of other kinds of ions than of krypton follows approximately the same process. It only must be noted that pure diffusion can be zero and sputtering of already collected atoms must be taken into account, when q/M_1 becomes an appreciable fraction of d_{12}/M_2 . The process can still be represented by a term D_i , now having a different meaning. Naturally many combinations of diffusion and sputtering can be responsible for the saturation phenomenon in a material.

6.2. Collection at low ion energy

In fig. 19 some q values are indicated to be very high. If the self-sputtering ratio is below one, the bombarding ions can build up a layer on the surface of the backing material. The self-sputtering ratio, S_e , is that measured, when ions impinge on a material of the same element as the ions. Building-up takes place at 45 keV energy on beryllium, boron carbon, magnesium, aluminium, silicon, calcium, scandium

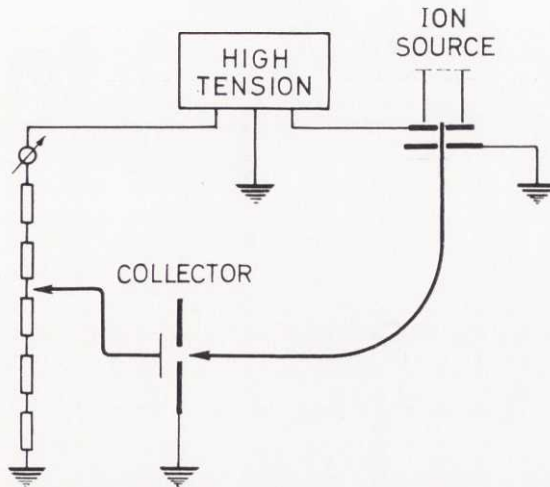


Fig. 23. Arrangement for ion retardation at the collector.

and titanium, impinging on a tantalum backing; for a few of these under special conditions regarding target temperature and current density.

The process of building-up indicates a method to produce thick targets by slowing down the ions to an energy below the point, where S_e is one. Slowing down can be made in the arrangement shown in fig. 23, where the collector is simply put on a high positive potential behind a defining slit. The entering beam is retarded to an adjustable rest energy of 1–5 keV. It is at the same time slightly defocused because the arrangement is acting as an electrostatic lens. Using a specially shaped electrode it is possible to get focusing instead of defocusing at the collector even at energies lower than 1 keV.¹¹⁾ We have not yet applied this arrangement, because we want the lines spread out uniformly over a small area. In fact we have used our linear sweep on the beam to produce uniform layers of 5–10 $\mu\text{g}/\text{cm}^2$ thickness.

The ion energy, where the selfsputtering is equal to one, has been calculated for a few elements in paper IV p. 287. These energies are very valuable for target preparation and are therefore given also here (table 2). Of course we must retard the ions to an energy well below that listed in table 2 in order to get a reasonable collection efficiency, which here is determined by the sputtering ratio. If the ion energy, where $S_e = 1$, is below 1–2 keV, S_e is approximately proportional to

TABLE 2
Ion energies for which the selfsputtering ratio is one.

Element	E (keV)
Fe	1.3
Ni	1.3
Cu	0.40
Zr	10.5
Nb	25
Mo	6.6
Ag	0.20
Sn	0.45
Cd	<0.05
Ta	3.7
W	3.0
Pt	0.5

the ion energy below 1–2 keV. A small “threshold energy” (the order of 50–100 eV), below which S_e is approximately zero can, however, sometimes be taken into account. In a few cases it is impossible to operate at the necessary low rest energy and thick samples cannot be prepared by the direct collection method, e.g., this must happen with sulfur, zinc, cadmium and probably with a few more which thus must be regarded as gases and collected at high ion energy only, with the collected amount limited to a few $\mu\text{g}/\text{cm}^2$.

An isotopic sample of thickness 50–200 $\mu\text{g}/\text{cm}^2$ is here regarded as a thick sample. If layers are wanted of this thickness or more, care must be taken, at the preparation, since a deposited thick layer may suddenly be destroyed by the beam, if it has a low thermal conductivity. In addition to the power dissipated by the ions on the collector a considerable electron current and power is always dissipated there. The electrons are produced by ion collisions with atoms in the rest gas and secondary emitted electrons from the rest of the collector are also present. Therefore the collector is heated up much more than expected from the measured current, which is the difference between the ion current and the electron current. Cooling may be necessary and also shielding because of the produced x-radiation. The physical properties of the deposited layer may also be different from the properties of bulk material and its S_e value, expected to be well

below one, instead is above one, and the layer is sputtered away. It is always the best to spread the beam out uniformly over the target area in order to avoid local heating up and eventual damage. This may also be the best condition for the further use of the sample. Vacuum conditions can probably also influence a building-up process. However, nothing is known about this at the moment.

Sometimes target preparation is made on very thin backing foils of a few $\mu\text{g}/\text{cm}^2$. Here a retarded ion beam must be used, otherwise the foil is burnt away already at a quite low current density.

The introduction of the retardation method has greatly improved the collection technique in a laboratory isotope separator. In our machine stable isotopes are nowadays always collected with a retarded ion beam, which permits the preparation of samples of an approximately known thickness. Furthermore the useful atoms are deposited as a surface layer on the backing material, not spread out within the large penetration depth in the material as for high energy ions. Even gas targets have been prepared at low ion energy, 2–5 keV, since it has been proved that the concentration of collected gas atoms is independent of the energy down to a few keV. Radioactive samples can also with advantage be prepared at a low energy in order to decrease e.g. absorption of beta particles in the backing material to a minimum. What is gained here can, however, be the subject of a large investigation of its own.

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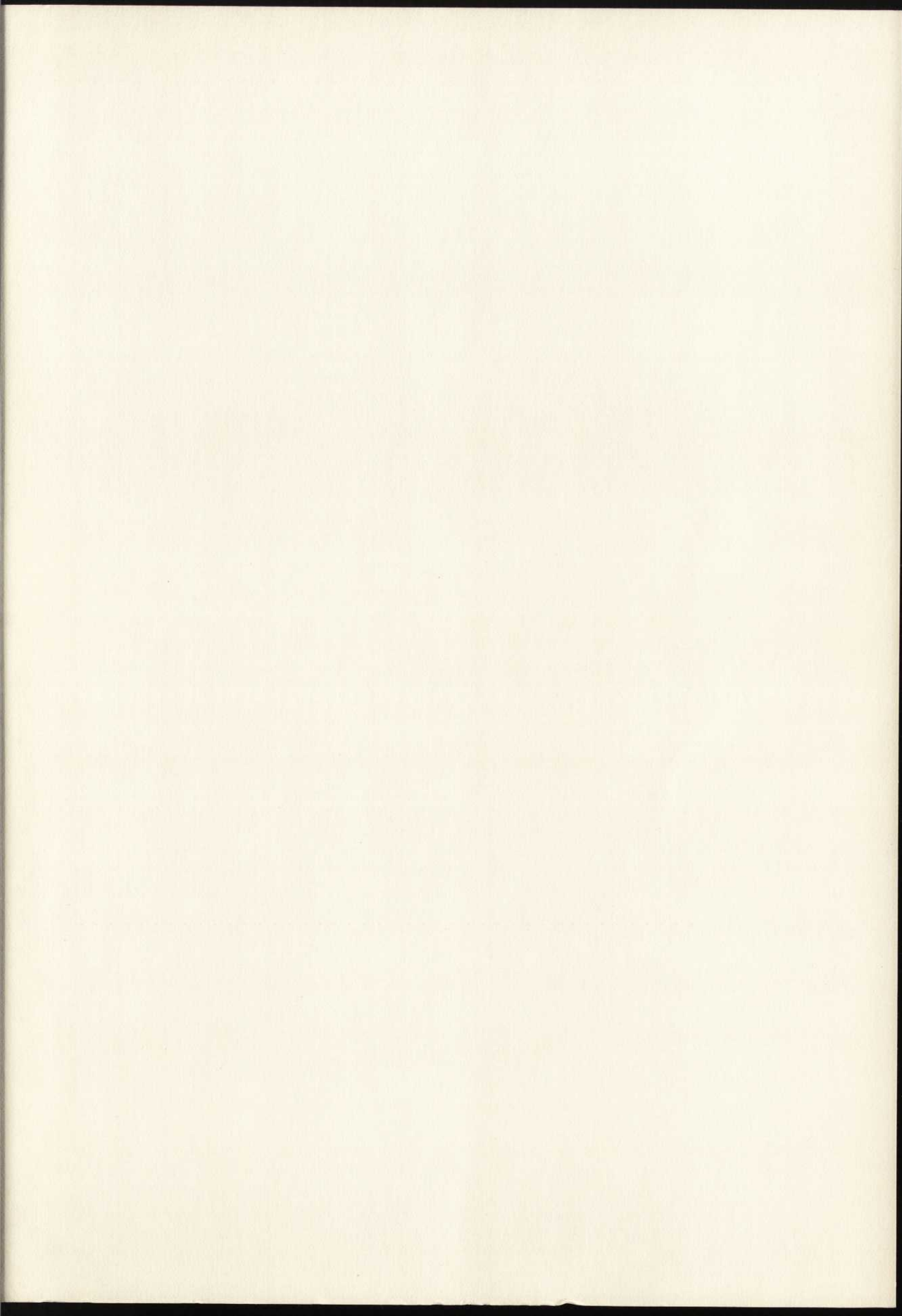
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